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(54) Title: CARBON BLACKS (57) Abstract Novel carbon blacks that impart advantageous properties to rubber and plastic compositions and may be utilized in place of lamp blacks, thermal carbon blacks and blends of carbon blacks. Also disclosed are novel rubber and plastic compositions incorporating the novel carbon blacks which exhibit advantageous combinations of compound processing and physical performance properties.		

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CARBON BLACKS

This application is a continuation-in-part of United States Patent Application Serial Number 07/935,794, Filed August 27, 1992.

5 FIELD OF THE INVENTION

The present invention relates to new carbon blacks which are suitable for various applications and particularly well suited for use in plastic and rubber compositions.

BACKGROUND

10 Carbon blacks may be utilized as pigments, fillers, reinforcing agents, and for a variety of other applications. They are widely used in the preparation of rubber compositions and plastic compositions where it is desirable to achieve an optimal combination of compound processing characteristics and
15 physical properties of fabricated parts.

Carbon blacks are generally characterized on the basis of their properties including, but not limited to, their surface areas, surface chemistry, aggregate sizes, and particle sizes. The properties of carbon blacks are
20 analytically determined by tests known to the art, including iodine adsorption number (I_2 No.), dibutyl phthalate adsorption (DBP), Tint value (TINT), Dst, Dmode and M-Ratio that is defined as the median Stokes diameter divided by the mode Stokes diameter ($M\text{-Ratio} = D_{\text{dst}}/D_{\text{mode}}$).

25 From the prior art several references are known. These include U.S. Patent No. 4,366,139; U.S. Patent No. 4,221,772; U.S. Patent No. 3,799,788; U.S. Patent No. 3,787,562; Soviet Union 1279991; Canadian 455504; Japanese 61-047759; British 1022988; and Japanese 61-283635. None of the aforementioned
30 references disclose the carbon black products of the present invention. Moreover, none of the aforementioned references describe the use for which the carbon blacks of the present invention are intended.

SUMMARY OF THE INVENTION

We have discovered nine new classes of carbon blacks advantageous for use in rubber and plastic compositions where compound processing and physical properties such as mixing energy, viscosity, cure rate, extrusion shrinkage, tensile, fatigue life, compression set, hardness, resistivity and surface appearance are important. These carbon blacks have been found to provide unique combinations of properties that make them especially well suited for use in extrusion, molded part, hose and belt applications.

The first class of carbon blacks has an Iodine adsorption number (I_2 No.) of 17-23 mg/g (milligrams I_2 per gram carbon black) and a DBP (dibutyl phthalate value) of 115-150 cc/100g (cubic centimeters of dibutyl phthalate per 100 grams carbon black). Preferably this class of carbon blacks is characterized by having an I_2 No. of about 20 mg/g.

The second class of carbon blacks has an I_2 No. of 10-19 mg/g and a DBP of 70-95 cc/100g.

The third class of carbon blacks has an I_2 No. of 12-20 mg/g and a DBP of 34-65 cc/100g. Preferably this class of carbon blacks is characterized by having an I_2 No. of 14-18 mg/g and a DBP of 36-55 cc/100g, and more preferably a DBP of either 36-42 cc/100g or 45-55 cc/100g.

The fourth class of carbon blacks has an I_2 No. of 28-43 mg/g, and a DBP of 28-47 cc/100g. Preferably this class of carbon blacks is characterized by having an I_2 No. of 30-42mg/g.

The fifth class of carbon blacks has an I_2 No. of 8-32 mg/g, a DBP of 28-150 cc/100g and an M-Ratio of 1.25-2.00.

The sixth class of carbon blacks has an I_2 No. of 33-70 mg/g, a DBP of 28-60 cc/100g and an M-Ratio of 1.25-2.00.

The seventh class of carbon blacks has an I_2 No. of 42-50 mg/g, a DBP of 61-105 cc/100g and an M-Ratio of 1.25-2.00.

The eighth class of carbon blacks has an I_2 No. of 51-62 mg/g, a DBP of 61-125 cc/100g and an M-Ratio of 1.25-2.00.

The ninth class of carbon blacks has an I_2 No. of 63-70

mg/g, a DBP of 61-105 cc/100g and an M-Ratio of 1.25-2.00.

We have also discovered new classes of rubber and plastic compositions containing the carbon blacks.

5 The carbon blacks of the present invention may be produced in a furnace carbon black reactor having a combustion zone, a transition zone, and a reaction zone. A carbon black yielding feedstock is injected into a hot combustion gas stream. The resultant mixture of hot combustion gases and feedstock passes into the reaction zone. Pyrolysis of the
10 carbon black yielding feedstock is stopped by quenching the mixture after the carbon blacks of the present invention have been formed. Preferably pyrolysis is stopped by injecting a quenching fluid. The process for preparing the novel carbon blacks of the present invention will be described in greater
15 detail hereinafter.

The rubbers and plastics for which the novel carbon blacks of this invention are effective include natural and synthetic rubbers and plastics. Generally, amounts of the carbon black product ranging from about 10 to about 300 parts
20 by weight can be used for each 100 parts by weight of rubber or plastic.

Among the rubbers or plastics suitable for use with the present invention are natural rubber, synthetic rubber and their derivatives such as chlorinated rubber; copolymers of
25 from about 10 to about 70 percent by weight of styrene and from about 90 to about 30 percent by weight of butadiene such as copolymer of 19 parts styrene and 81 parts butadiene, a copolymer of 30 parts styrene and 70 parts butadiene, a copolymer of 43 parts styrene and 57 parts butadiene and a
30 copolymer of 50 parts styrene and 50 parts butadiene; polymers and copolymers of conjugated dienes such as polybutadiene, polyisoprene, polychloroprene, and the like, and copolymers of such conjugated dienes with an ethylenic group-containing monomer copolymerizable therewith such as styrene, methyl
35 styrene, chlorostyrene, acrylonitrile, 2-vinyl-pyridine, 5-methyl-2-vinylpyridine, 5-ethyl-2-vinylpyridine, 2-methyl-5-vinylpyridine, alkyl-substituted acrylates, vinyl ketone, methyl isopropenyl ketone, methyl vinyl ether,

alphanethylene carboxylic acids and the esters and amides thereof such as acrylic acid and dialkylacrylic acid amide; also suitable for use herein are copolymers of ethylene and other high alpha olefins such as propylene, butene-1 and 5 penetene-1; particularly preferred are the ethylene-propylene copolymers wherein the ethylene content ranges from 20 to 90 percent by weight and also the ethylene-propylene polymers which additionally contain a third monomer such as dicyclopentadiene, 1,4-hexadiene and methylene norbornene. 10 Additionally preferred polymeric compositions are olefins such as polypropylene and polyethylene.

An advantage of the carbon blacks of the present invention is that the carbon blacks are useful for incorporation into natural rubbers, synthetic rubbers, 15 plastics or blends thereof for industrial applications, particularly where compound processing and part performance characteristics are important.

A further advantage of the carbon blacks of the present invention is that certain of the carbon blacks of the present 20 invention will replace blends of carbon blacks in applications that currently require the use of blends of carbon blacks to achieve desired performance characteristics.

Other advantages of the present invention will become apparent from the following more detailed description of the 25 invention.

BRIEF DESCRIPTION OF THE DRAWINGS:

Figure 1 is a cross-sectional view of a portion of one type of furnace carbon black reactor which may be utilized to produce the carbon blacks of the present invention.

5 Figure 2 is a sample histogram of the weight fraction of the aggregates of a carbon black sample versus the Stokes Diameter in a given sample.

DETAILED DESCRIPTION OF THE INVENTION

10 The analytical properties of each of the nine classes of carbon blacks of the present invention are set forth in Table 1 below:

Table 1.

Class	I ₂ No.	DBP	M-Ratio
15 First First Preferred	17-23 20	115- 150 115- 150	--- ---
Second	10-19	70-95	---
20 Third Third Preferred Third More Preferred Third More Preferred	12-20 14-18 14-18 14-18	34-65 36-55 36-42 45-55	--- --- --- ---
25 Fourth Fourth Preferred	28-43 30-42	28-47 28-47	--- ---
Fifth	8-32	28- 150	1.25- 2.00
Sixth	33-70	28-60	1.25- 2.00
30 Seventh	42-50	61- 105	1.25- 2.00
Eighth	51-62	61- 125	1.25- 200
Ninth	63-70	61- 105	1.25- 2.00

The carbon blacks of the present invention may be produced in a modular, also referred to as "staged", furnace carbon black reactor. A section of a typical modular furnace carbon black reactor which may be utilized to produce the carbon black of the present invention is depicted in Figure 1. Other details of a typical modular furnace carbon black reactor may be found, for example, in the description contained in U.S. Patent No. 3,922,335, the disclosure of which is herein incorporated by reference. A carbon black reactor particularly well-suited for the production of the carbon blacks of the present invention is described in commonly assigned U.S. patent application serial number 07/818,943, filed January 10, 1992, the disclosure of which is hereby incorporated by reference. The carbon blacks of the Examples described herein were made by the process described in the '943 application.

The '943 application describes a process for producing carbon blacks wherein auxiliary hydrocarbon is added to the reaction zone of a multistage reactor and the primary combustion and overall combustion of the reaction are adjusted so that the SSI of the process is less than zero. The SSI of the process may be determined by the following relationships:

$$SSI = \frac{SAS_{mf} - SAS_{ah}}{|SAS_{mf}|} \quad (1)$$

where

$$SAS_{mf} = \frac{\Delta (DBP)_{mf}}{\Delta (IodineNumber)_{mf}} ; SAS_{ah} = \frac{\Delta (DBP)_{ah}}{\Delta (IodineNumber)_{ah}} \quad (2)$$

$|SAS_{mf}|$ = Absolute value of SAS_{mf} ;

$\Delta (DBP)_{mf}$ = the change in DBPA of the carbon black due to a change in feedstock flow rate while all other process operating conditions are held constant;

$\Delta (Iodine\ Number)_{mf}$ = the change in iodine adsorption number of the carbon black due to a change in feedstock flow rate while all other process operating conditions are held constant;

$\Delta(\text{DBP})_{\text{ah}} =$ the change in DBPA of the carbon black due to a change in auxiliary hydrocarbon flow rate while all other process operating conditions are held constant; and

5 $\Delta(\text{Iodine Number})_{\text{ah}} =$ the change in iodine adsorption number of the carbon black due to a change in auxiliary hydrocarbon flow rate while all other process operating conditions are held constant.

10

The "auxiliary hydrocarbon" comprises hydrogen or any hydrocarbon having a molar hydrogen-to-carbon ratio greater than the molar hydrogen-to-carbon ratio of the feedstock.

15 Referring to Figure 1, the carbon blacks of the present invention may be produced in a furnace carbon black reactor 2, having a combustion zone 10, which has a zone of converging diameter 11, transition zone 12, entry section 18, and reaction zone 19. The diameter of the combustion zone 10, up to the point where the zone of converging diameter 11 begins, is shown as D-1; the diameter of zone 12, as D-2; the diameters of the stepped entry section, 18, as D-4, D-5, D-6, and D-7; and the diameter of zone 19, as D-3. The length of the combustion zone 10, up to the point where the zone of converging diameter 11 begins, is shown as L-1; the length of the zone of converging diameter is shown as L-2; the length of the transition zone is shown as L-3; and the lengths of the steps in the reactor entry section, 18, as L-4, L-5, L-6 and L-7.

20

25

30 To produce carbon blacks, hot combustion gases are generated in combustion zone 10, by contacting a liquid or gaseous fuel with a suitable oxidant stream such as air, oxygen, mixtures of air and oxygen or the like. Among the fuels suitable for use in contacting the oxidant stream in combustion zone 10 to generate the hot combustion gases are any of the readily combustible gas, vapor, or liquid streams such as natural gas, hydrogen, carbon monoxide, methane, acetylene, alcohol, or kerosene. It is generally preferred, however, to utilize fuels having a high content of carbon-containing

35

components and in particular, hydrocarbons. The ratio of air to natural gas utilized to produce the carbon blacks of the present invention may preferably be from about 10:1 to about 100:1. To facilitate the generation of hot combustion gases, the oxidant stream may be preheated.

The hot combustion gas stream flows downstream from zones 10 and 11 into zones 12, 18, and 19. The direction of the flow of hot combustion gases is shown in the figure by the arrow. Carbon black-yielding feedstock 30 is introduced at point 32 (located in zone 12), and/or at point 70 (located in zone 11). Suitable for use herein as carbon black-yielding hydrocarbon feedstocks, which are readily volatilizable under the conditions of the reaction, are unsaturated hydrocarbons such as acetylene; olefins such as ethylene, propylene, butylene; aromatics such as benzene, toluene and xylene; certain saturated hydrocarbons; and other hydrocarbons such as kerosenes, naphthalenes, terpenes, ethylene tars, aromatic cycle stocks and the like.

The distance from the end of the zone of converging diameter 11 to point 32 is shown as F-1. Generally, carbon black-yielding feedstock 30 is injected in the form of a plurality of streams which penetrate into the interior regions of the hot combustion gas stream to insure a high rate of mixing and shearing of the carbon black-yielding feedstock by the hot combustion gases so as to rapidly and completely decompose and convert the feedstock to carbon black.

Auxiliary hydrocarbon is introduced at point 70 through probe 72 or through auxiliary hydrocarbon passages 75 in the walls which form the boundaries of zone 12 of the carbon black forming process or through auxiliary hydrocarbon passages 76 in the walls which form the boundaries of zones 18 and/or 19 of the carbon black forming process. The auxiliary hydrocarbon may be introduced at any location between the point immediately after the initial combustion reaction of the first-stage fuel and the point immediately before the end of formation of carbon black provided that unreacted auxiliary hydrocarbon eventually enters

the reaction zone.

The distance from point 32 to point 70 is shown as H-1.

In the Examples described herein, the auxiliary hydrocarbon was introduced through either three or four orifices in the same axial plane as the carbon black yielding feedstock streams. Th
5 orifices are arranged in an alternating pattern, one feedstock, the next auxiliary hydrocarbon, spaced evenly around the outer periphery of section 12. As will be noted, however, this is merely exemplary and is not intended to be limiting of the
10 methods usable for introducing auxiliary hydrocarbon.

The mixture of carbon black-yielding feedstock and hot combustion gases flows downstream through zone 12 into zone 18 and then into zone 19. Quench 60, located at point 62, injecting quenching fluid 50, which may be water, is utilized to
15 stop chemical reaction when carbon blacks are formed. Point 62 may be determined in any manner known to the art for selecting the position of a quench to stop pyrolysis. One method for determining the position of the quench to stop pyrolysis is by determining the point at which an acceptable toluene extract
20 level for the carbon black is reached. Toluene extract level may be measured by using ASTM Test D1618-83 "Carbon Black Extractables - Toluene Discoloration". Q is the distance from the beginning of zone 18 to quench point 62, and will vary according to the position of Quench 60.

25 After the mixture of hot combustion gases and carbon black-yielding feedstock is quenched, the cooled gases pass downstream into any conventional cooling and separating means whereby the carbon blacks are recovered. The separation of th carbon black from the gas stream is readily accomplished by
30 conventional means such as a precipitator, cyclone separator or bag filter. This separation may be followed by pelletizing using, for example, a wet pelletizer.

The following testing procedures are used in evaluating the analytical and physical properties of the carbon blacks of the
35 present invention.

Iodine adsorption number of the carbon blacks (I_2 No.) was determined according to ASTM Test Procedure D 1510. Tinting strength (Tint) of the carbon blacks was determined according to ASTM Test Procedure D3265-85a. The DBP (dibutyl phthalat value) of the carbon blacks was determined according to the
5 procedure set forth in ASTM D3493-86. The cetyl-trimethyl ammonium bromide absorption value (CTAB) of the carbon blacks was determined according to ASTM Test Procedure D3765-85.

Dmode, and Dst of the carbon blacks were determined from a
10 histogram of the weight fraction of carbon black versus th Stokes diameter of the carbon black aggregates, as shown in Figure 2. The data used to generate the histogram are determined by the use of a disk centrifuge such as the one manufactured by Joyce Loebel Co. Ltd. of Tyne and Wear, Unit d
15 Kingdom. The following procedure is a modification of the procedure described in the instruction manual of the Joyce Loebel disk centrifuge file reference DCF 4.008 published on February 1, 1985, the teachings of which are hereby incorporated by reference, and was used in determining the data.

20 The procedure is as follows. 10 mg (milligrams) of a carbon black sample are weighed in a weighing vessel, th n added to 50 cc of a solution of 10% absolute ethanol and 90% distilled water which is made 0.05% NONIDET P-40 surfactant (NONIDET P-40 is a registered trademark for a surfactant manufactured and sold
25 by Shell Chemical Co.). The resulting suspension is dispersed by means of ultrasonic energy for 15 minutes using Sonifier Model No. W 385, manufactured and sold by Heat Systems Ultrasonics Inc., Farmingdale, New York.

Prior to the disk centrifuge run the following data ar
30 entered into the computer which records the data from the disk centrifuge:

1. The specific gravity of carbon black, taken as 1.86 g/cc;
2. The volume of the solution of the carbon black
35 dispersed in a solution of water and ethanol, which in this

instance is 0.5 cc.;

3. The volume of spin fluid, which in this instance is 10 cc of water;

4. The viscosity of the spin fluid, which in this instance is taken as 0.933 centipoise at 23 degrees C;

5. The density of the spin fluid, which in this instance is 0.9975 g/cc at 23 degrees C;

6. The disk speed, which in this instance is 8000 rpm;

7. The data sampling interval, which in this instance is 1 second.

The disk centrifuge is operated at 8000 rpm while the stroboscope is operating. 10 cc of distilled water are injected into the spinning disk as the spin fluid. The turbidity level is set to 0; and 1 cc of the solution of 10% absolute ethanol and 90% distilled water is injected as a buffer liquid. The cut and boost buttons of the disk centrifuge are then operated to produce a smooth concentration gradient between the spin fluid and the buffer liquid and the gradient is monitored visually. When the gradient becomes smooth such that there is no distinguishable boundary between the two fluids, 0.5 cc of the dispersed carbon black in aqueous ethanol solution is injected into the spinning disk and data collection is started immediately. If streaming occurs the run is aborted. The disk is spun for 20 minutes following the injection of the dispersed carbon black in aqueous ethanol solution. Following the 20 minutes of spinning, the disk is stopped, the temperature of the spin fluid is measured, and the average of the temperature of the spin fluid measured at the beginning of the run and the temperature of the spin fluid measured at the end of the run is entered into the computer which records the data from the disk centrifuge. The data is analyzed according to the standard Stokes equation and is presented using the following definitions:

Carbon black aggregate - a discrete, rigid colloidal entity that is the smallest dispersible unit; it is composed of

extensively coalesced particles;

Stokes diameter - the diameter of a sphere which sediments in a viscous medium in a centrifugal or gravitational field according to the Stokes equation. A non-spherical object, such as a carbon black aggregate, may also be represented in terms of the Stokes diameter if it is considered as behaving as a smooth, rigid sphere of the same density, and rate of sedimentation as the object. The customary units are expressed in nanometer diameters.

Mode (Dmode for reporting purposes) - The Stokes diameter at the point of the peak (Point A of Figure 2 herein) of the distribution curve for Stokes diameter.

Median Stokes diameter - (D₅₀ for reporting purposes) the point on the distribution curve of Stokes diameter where 50% by weight of the sample is either larger or smaller. It therefore represents the median value of the determination.

The modulus, tensile and elongation of the EPDM compositions were measured by the procedure set forth in ASTM D412-87.

The Shore A Hardness of the EPDM compositions was determined according to the procedure set forth in ASTM D-2240-86.

Rebound data for the EPDM compositions was determined according to the procedure set forth in ASTM D1054, utilizing a ZWICK Rebound Resilience Tester, Model 5109, manufactured by Zwick of America, Inc., Post Office Box 997, East Windsor, Connecticut 06088. Instructions for determining the rebound values accompany the instrument.

The Compression set of the EPDM compositions was determined according to the procedure set forth in ASTM D395, wherein the composition was tested at 150° F for 70 hours.

The extrusion shrinkage of the EPDM compositions was determined by the procedure set forth in ASTM D-3674. The extrusion shrinkage was measured on the BRABENDER extruder at 100° C and 50 rpm using a 5 mm diameter die.

The viscosity of the EPDM compositions was determined by the procedure set forth in ASTM D-1646 using a Monsanto MPT capillary rheometer maintained at 100° C using a die having a ratio of $L/D'=16$ and $D=0.0787\text{mm}$ (millimeter). The shear rate
5 ranged from 10 to 150 1/seconds.

Mixing energy is the total amount of energy put into the compositions which is determined by integrating the mixing torque curve over the course of the mixing cycle, described hereinafter.

10 Cure characteristics of the EPDM compositions were measured using a Monsanto MDR curemeter maintained at 160° C. The time to reach 90% cure reaction (t'_{90}), the total torque change during cure reaction (ΔL) and cure rate index (CRI; $(\text{CRI} = 1/(t'_{90} - t_{s1}) \times 100)$ where t_{s1} = the time when the torque level
15 is 1 unit above minimum torque (t_{s1} is also referred to as scorch time)) are reported for the example EPDM compositions. The tests were conducted according to the instructions furnished with the Monsanto MDR curemeter.

Composition resistivity was measured on plaque samples 2
20 inches wide by 6 inches long by 0.085 inch thick. The plaques were painted on both ends with a half inch width of silver paint. The sample was conditioned to produce a stable reading by cycling from room temperature to 100° C and back to room temperature, followed by aging at 90° C for 24 hours. The
25 stabilized resistivity was measured at the end of the aging cycle, and once again after the sample was allowed to cool back to room temperature.

The effectiveness and advantages of the present invention will be further illustrated by the following examples.

30 EXAMPLES 1-8

Examples of the novel carbon blacks of the present invention were prepared in a reactor generally described herein, and as depicted in Figure 1, utilizing the reactor conditions and geometry set forth in Table 3. The fuel utilized in the

combustion reaction was natural gas. The auxiliary hydrocarbon used was also natural gas. The liquid feedstock utilized had the properties indicated in Table 2 below:

Table 2

		FEEDSTOCK PROPERTIES
5	Hydrogen/Carbon Ratio	0.93
	Hydrogen (wt%)	7.19
	Carbon (wt%)	92.1
	Sulfur (wt%)	0.3
10	Nitrogen (wt%)	0.41
	A.P.I. Gravity 15.6/15.6° C (60/60° F) [ASTM D-287]	-1.6
15	Specific Gravity 15.5/15.6° C (60/60 °F) [ASTM D-287]	1.092
20	Viscosity, SUS (54.4°C) m ² /s [ASTM D-88]	2.6×10^{-5}
	Viscosity, SUS (98.9°C) m ² /s [ASTM D-88]	5.2×10^{-6}
25	BMCI (Visc-Grav)	133

The reactor conditions and geometry were as set forth in Table 3 below.

Tabl 3 Reactor Geometry and Operating Conditions.

Example No.	1	2	3	4	5	6	7	8
5								
D-1 (m)	0.18	0.18	0.18	0.18	0.18	0.18	0.18	0.18
D-2 (m)	0.11	0.11	0.11	0.11	0.13	0.11	0.13	0.13
D-3 (m)	0.69	0.69	0.69	0.69	0.91	0.69	0.69	0.69
D-4 (m)	0.25	0.25	0.25	0.25	0.26	0.25	0.25	0.36
D-5 (m)	0.69	0.69	0.69	0.69	0.33	0.69	0.69	0.69
D-6 (m)	0.69	0.69	0.69	0.69	0.91	0.69	0.69	0.69
10								
D-7 (m)	0.69	0.69	0.69	0.69	0.91	0.69	0.69	0.69
L-1 (m)	0.61	0.61	0.61	0.61	0.61	0.61	0.61	0.61
L-2 (m)	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30
L-3 (m)	0.23	0.23	0.23	0.23	0.23	0.23	0.23	0.23
L-4 (m)	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.14
15								
L-5 (m)	0.10	0.10	0.10	0.10	0.23	0.10	0.10	0.10
L-6 (m)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
L-7 (m)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
F-1 (m)	0.11	0.11	0.11	0.11	0.11	0.11	0.11	0.11
Q (m)	7.93	7.93	7.93	7.93	7.93	7.93	19.8	7.93
20								
Combustion Air (SCMS)	0.373	0.634	0.634	0.634	0.634	0.634	0.448	0.634
Comb. Air Preheat (K)	755	755	755	755	755	755	755	755
25								
Burgen Mat. Gas (10 ² x SCMS)	0.9	2.0	2.0	2.0	1.8	2.5	1.4	2.0
Feedstock Injection Orifice Dia. (cm)	0.198	0.218	0.198	0.198	0.185	0.198	0.198	0.206
30								
No. Feedstock Injection Orifices	3	3	3	3	4	3	4	4
Feedstock Rate (10 ² x m ³ /s)	1.3	2.1	1.9	2.3	1.3	1.9	1.6	2.1
35								
Feedstock Temp. (K)	483	463	451	438	467	439	467	415
K ⁺ Conc. (g/m ³)	0.0	87.1	81.0	0.0	388.0	11.4	0.8	20.3
40								
Aux. HC Injection Orifice Diameter (cm)	0.635	0.635	0.635	0.635	0.635	0.635	0.635	0.635
No. Aux. HC Injection Orifices (*)	3	3	3	3	4	3	4	4
45								
Aux. HC Rate (10 ² x SCMS)	2.4	3.5	2.1	3.9	3.1	2.1	1.7	0.0
Primary Combustion (%)	400	400	400	400	300	325	400	400
50								
Overall Combustion (%)	21.7	22.5	25.4	21.5	22.9	25.5	21.4	25.8

(*) - The feedstock and auxiliary hydrocarbon orifices were arranged in the same axial plane in an alternating sequence around the periphery of the reactor.

55 HC = hydrocarbon

The carbon blacks produced in runs 1-8 were then analyzed according to the procedures described herein. The analytical properties of the carbon blacks were as set forth in Table 4.

Table 4 Carbon Black Analytical Properties

Example	1	2	3	4	5	6	7	8
I ₂ No. (mg/g)	22.1	13.6	28.0	16.3	40.7	28.8	28.7	57.9
DBP (cc/100g)	132.5	37.1	35.8	84.6	43.1	64.5	119.5	95.8
CTAB (m ² /g)	25.3	17.3	29.8	18.7	45.1	28.7	27.8	53.1
Tint (5)	33.0	33.4	54.7	37.5	85.6	45.7	33.5	74.2
Dmode (nm)	289	246	212	276	176	212	279	140
Dst (nm)	459	390	300	492	240	312	486	180
M-Ratio	1.59	1.58	1.42	1.78	1.36	1.47	1.74	1.29

These carbon blacks and four control carbon blacks were utilized in the following examples. The four control carbon blacks utilized, A-D, had the analytical properties shown below in Table 5:

Table 5 Control Carbon Black Analytical Properties

Control	A	B	C	D
Type	GPF	FEF	Therma 1	SRF
I ₂ No. (mg/g)	35.4	40.0	8.2	29.9
DBP (cc/100g)	91.0	111.7	37.5	68.5
CTAB (m ² /g)	35.9	44.3	9.9	30.1
Tint (%)	57.8	64.6	21.7	51.6
Dmode (nm)	206	144	416	256
Dst (nm)	220	186	492	288
M-Ratio	1.07	1.29	1.18	1.12

GPF = general purpose furnace

FEF = fast extruding furnace

Thermal = carbon black produced by a thermal process

SRF = semi reinforcing furnace

EXAMPLE 9

The carbon blacks of the present invention produced in example runs 1-8 were incorporated into EPDM (ethylene-propylene diene polymethylene) compositions and compared to EPDM compositions incorporating the four control carbon blacks. The EPDM compositions were prepared utilizing each of the carbon black samples in an amount of 200 parts by weight in the EPDM Composition Formulation shown below in Table 6.

Table 6 - EPDM COMPOSITION FORMULATION

INGREDIENT	PART BY WEIGHT
EPDM	100
Carbon Black	200
Sunpar 2280 Oil	100
Zinc Oxide	5
Stearic Acid	1
TMTDS	2.7
Butyl Zimate	2.7
Methyl Zimate	2.7
Sulfur	0.5
Sulfasan R	1.7

EPDM - EXXON VISTALON®5600, manufactured and sold by EXXON Corporation, Houston, Texas;
Sunpar 2280 - A trademarked oil manufactured and sold by Sun Oil Company;
TMTDS - Tetramethylthiuram disulfide;
Butyl Zimate - A trademarked zinc dibutyldithiocarbamate manufactured and sold by R. T. Vanderbilt Co.;
Methyl Zimate - A trademarked zinc dimethyldithiocarbamate manufactured and sold by R. T. Vanderbilt Co.;
Sulfasan R - A trademarked 4,4'-dithiodimorpholine, manufactured and sold by Monsanto Co., St. Louis, Missouri

The EPDM compositions were produced as follows.
A Banbury BR mixer was started and maintained at a temperature of 45° C and a rotor speed of 77 RPM. EPDM was added to the mixer and mixed for approximately 30 seconds. The Sunpar 2280

oil, zinc oxide and stearic acid, were added to the EPDM and mixed for approximately 2 additional minutes. The carbon black was added to the mixture and the temperature of the mixing chamber was cooled and maintained at a temperature of below approximately 135° C. The carbon black containing EPDM mixture was mixed for approximately 4 1/2 minutes and then the curing agents, TMTDS, Butyl Zimate, Methyl Zimate, Sulfur and Sulfasan R, were added to the mixture. The resulting mixture was mixed for approximately 1 1/2 minutes while the temperature was maintained at below approximately 135° C. The batch composition was then discharged from the mixer and analyzed by the techniques described herein.

The EPDM compositions produced using the carbon blacks of the present invention produced in runs 1-8 described herein had the performance characteristics set forth below in Table 7.

The EPDM compositions incorporating the control carbon blacks A-D were also evaluated according to the procedures described herein. The results are set forth in Tables 8-13 below, where comparisons between EPDM compositions containing carbon blacks of the present invention and EPDM compositions containing the most appropriate control carbon black are made.

Table 7 Comparison of EPDM Composition Performance.

Example No.	1	2	3	4	5	6	7	8
Carbon Black Analytical Properties:								
5 Iodine Number (mg/g)	22.1	13.6	28.0	16.3	40.7	28.8	28.7	57.9
DBP (cc/100g)	132.5	37.1	35.8	84.6	43.1	64.5	119.5	95.8
CTAB (m ² /g)	25.3	17.3	29.8	18.7	45.1	28.7	27.8	53.1
Tint (%)	33.0	33.4	54.7	32.5	85.6	45.7	42.3	74.2
Mode Stokes Dia. (nm)	289	246	212	276	176	212	252	10
10 Median Stokes Dia. (nm)	459	390	300	492	240	312	348	180
M-Ratio (Median/Mode Stokes)	1.59	1.58	1.42	1.78	1.36	1.47	1.38	1.29
EPDM Composition Performance at 200 phr:								
15 Viscosity (Pa·S) @10 sec ⁻¹	16300	12100	10900	18000	14100	16000	22900	23700
Viscosity (Pa·S) @150 sec ⁻¹	2600	1540	1590	1920	1720	1790	2790	3130
Mixing Energy (MJ/m ³)	1300	918	876	1110	943	1139	1348	1571
20 Extrusion Rate (g/min)	37.0	35.3	42.3	40.0	34.8	34.2	47.1	50.6
Extrusion Shrinkage (%)	12.5	37.6	43.0	21.7	41.1	27.1	13.8	12.5
t ₉₀ (min)	8.23	11.8	11.9	9.7	9.6	10.2	10.5	9.9
ΔL (g·m)	238	234	206	272	271	247	308	218
25 Hardness (Shore A)	79	57	65	69	65	67	76	79
E100 (10 ⁴ x N/m ²)	662	184	175	392	192	299	540	628
Tensile (10 ⁴ x N/m ²)	1069	779	974	925	1009	977	929	1268
Elong. at Break (%)	243	592	623	389	595	397	232	237
Rebound (%)	39	51.7	43.7	47.4	34.2	44.2	40.0	30.5
30 Compression Set (%) (70 hrs, 150°C)	55	56	50	52	65	54	54	58
Resistivity (Ω·cm) @25°C	72	94800	---	106	208	83	24	---
35 Resistivity (Ω·cm) @90°C	127	18200	---	153	178	104	43	---

--- = not evaluated

Table 8. Comparison of EPDM Composition Performance

Example No.	1	7	Control B
Carbon Black Analytical Properties:			
Iodine Number (mg/g)	22.1	228.7	40.0
DBP (cc/100g)	132.5	119.5	111.7
CTAB (m ² /g)	25.3	27.8	44.3
Tint (%)	33.0	42.3	64.6
Mode Stokes Dia. (nm)	289	252	144
Median Stokes Dia. (nm)	459	348	186
M-Ratio (Median/Mode Stokes)	1.59	1.38	1.29
EPDM Composition Performance at 200 phr:			
Viscosity (Pa·S) @ 10 sec ⁻¹	16300	22900	30700
Viscosity (Pa·S) @ 150 sec ⁻¹	2600	2790	3450
Mixing Energy (MJ/m ³)	1300	1348	1561
Extrusion Rate (g/min)	37.0	47.1	44.7
Extrusion Shrinkage (%)	12.5	13.8	18.2
t'90 (min)	8.23	10.5	7.45
ΔL (g·m)	238	308	293
Hardness (Shore A)	79	76	82
E100 (10 ⁴ x N/m ²)	662	540	650
Tensile (10 ⁴ x N/m ²)	1069	929	1103
Elong. at Break (%)	243	232	184
Rebound (%)	39	40.0	31.0
Compression Set (%) (70 hrs, 150°C)	55	54	57
Resistivity (Ω·cm) @25°C	72	24	43
Resistivity (Ω·cm) @90°C	127	43	69

These results set forth in Table 8 indicate that at a carbon black level of 200 phr the EPDM compositions incorporating the carbon blacks of the present invention have lower viscosity and lower mixing energy. Therefore the EPDM compositions incorporating the carbon blacks of the present invention exhibit better processing characteristics than the EPDM compositions incorporating the control carbon blacks.

Table 9. Comparison of EPDM Composition Performance

Example No.	1	Control B
Carbon Black Analytical Properties:		
Iodine Number (mg/g)	22.1	40.0
DBP (cc/100g)	132.5	111.7
CTAB (m ² /g)	25.3	44.3
Tint (%)	33.0	64.6
Mode Stokes Dia. (nm)	289	144
Median Stokes Dia. (nm)	459	186
M-Ratio (Median/Mode Stokes)	1.59	1.29
EPDM Composition Performance at 150 phr:		
Viscosity (Pa·S) @ 10 sec ⁻¹	12500	22200
Viscosity (Pa·S) @ 150 sec ⁻¹	1900	1940
Mixing Energy (MJ/m ³)	1010	1222
Extrusion Rate (g/min)	39	37.4
Extrusion Shrinkage (%)	17.2	19.8
t'90 (min)	9.25	8.33
ΔL (g·m)	200	249
Hardness (Shore A)	69	71
E100 (10 ⁴ x N/m ²)	407	376
Tensile (10 ⁴ x N/m ²)	1145	1179
Elong. at Break (%)	436	370
Rebound (%)	46.1	35.8
Compression Set (%) (70 hrs, 150°C)	57	57
Resistivity (Ω·cm) @25°C	400	88
Resistivity (Ω·cm) @90°C	640	118

The results set forth in Table 9 show the advantage of utilizing a carbon black of the present invention in an EPDM composition for an application where resistivity is a critical requirement. As shown in the table, at 150 phr loading, which is typical for a cooling hose application, the EPDM composition incorporating the carbon black of the present invention exhibits a higher resistivity than the EPDM composition incorporating the control carbon black.

Additionally, the EPDM composition incorporating the carbon black of the present invention has lower viscosity and lower mixing energy than the EPDM composition incorporating the control carbon black. This indicates that EPDM compositions incorporating the carbon black of the present invention will have improved processibility in comparison with EPDM compositions incorporating the control carbon black.

Table 10 Comparison of EPDM Composition Performance.

Example No.	6	Control D
Carbon Black Analytical Properties:		
Iodine Number (mg/g)	28.8	29.9
DBP (cc/100g)	64.5	68.5
CTAB (m ² /g)	28.7	30.1
Tint (%)	45.7	51.6
Mode Stokes Dia. (nm)	212	256
Median Stokes Dia. (nm)	312	288
M-Ratio (Median/Mode Stokes)	1.47	1.12
EPDM Composition Performance at 200 phr:		
Viscosity (Pa·S) @ 10 sec ⁻¹	16000	16600
Viscosity (Pa·S) @ 150 sec ⁻¹	1790	1880
Mixing Energy (MJ/m ³)	1139	1091
Extrusion Rate (g/min)	34.2	36.8
Extrusion Shrinkage (%)	27.1	23.8
t'90 (min)	10.2	11.2
ΔL (g·m)	247	270
Hardness (Shore A)	67	69
E100 (10 ⁴ x N/m ²)	299	338
Tensile (10 ⁴ x N/m ²)	9775	9899
Elong. at Break (%)	397	421
Rebound (%)	44.2	40.6
Compression Set (%) (70 hrs, 150°C)	54	57

The results set forth in Table 10 indicate that the compression set of the EPDM composition incorporating the carbon black of the present invention is lower than the compression set of the EPDM composition incorporating the control carbon black. Thus an EPDM composition incorporating the carbon black of the present invention is more resistant to permanent deformation. As a result the compositions will be particularly advantageous for use in sealing applications such as door seals and weather stripping.

Table 11 - Comparison of EPDM Composition Performance

Example No.	2	Control C
Carbon Black Analytical Properties:		
Iodine Number (mg/g)	13.6	8.2
DBP (cc/100g)	371.	37.5
CTAB (m ² /g)	17.3	9.9
Tint (%)	33.4	21.7
Mode Stokes Dia. (nm)	246	416
Median Stokes Dia. (nm)	390	492
M-Ratio (Median/Mode Stokes)	1.58	1.18
EPDM Composition Performance at 200 phr:		
Viscosity (Pa·S) @ 10 sec ⁻¹	12100	10400
Viscosity (Pa·S) @ 150 sec ⁻¹	1540	1490
Mixing Energy (MJ/m ³)	918	799
Extrusion Rate (g/min)	35.3	32.0
Extrusion Shrinkage (%)	37.6	43.5
t'90 (min)	11.8	13.2
ΔL (g·m)	234	220
Hardness (Shore A)	57	53
E100 (10 ⁴ x N/m ²)	184	109
Tensile (10 ⁴ x N/m ²)	779	933
Elong. at Break (%)	592	794
Rebound (%)	51.7	55.0
Compression Set (%) (70 hrs, 150°C)	56	54

The results set forth in Table 11 indicate that the carbon blacks of the present invention provides improved reinforcement properties, particularly higher modulus and higher hardness to EPDM compositions when compared to thermal carbon blacks. Thus the carbon blacks of the present invention advantageously provide furnace carbon black replacements for thermal carbon blacks or carbon black blends containing thermal carbon blacks.

Table 12 Comparison of EPDM Composition Performance.

Example No.	3	4	5	Contr l C	Contr l D
Carbon Black Analytical Properties:					
Iodine Number (mg/g)	28.0	16.3	40.7	8.2	29.9
DBP (cc/100g)	35.8	84.6	43.1	37.5	68.5
CTAB (m ² /g)	29.8	18.7	45.1	9.9	30.1
Tint (%)	54.7	32.5	85.6	21.7	51.6
Mode Stokes Dia. (nm)	212	276	176	416	256
Median Stokes Dia. (nm)	300	492	240	492	288
M-Ratio (Median/Mode Stokes)	1.42	1.78	1.36	1.18	1.12
EPDM Composition Performance at 200 phr:					
Viscosity (Pa·S) @ 10 sec ⁻¹	10900	18000	14100	10400	16600
Viscosity (Pa·S) @ 150 sec ⁻¹	1590	1920	1720	1490	1880
Mixing Energy (MJ/m ³)	876	1110	943	799	1091
Extrusion Rate (g/min)	42.3	40.0	34.8	32.0	36.8
Extrusion Shrinkage (%)	43.0	21.7	41.1	43.5	23.8
t' ₉₀ (min)	11.9	9.7	9.6	13.2	11.2
ΔL (g·m)	206	272	271	220	270
Hardness (Shore A)	65	69	65	53	69
E100 (10 ⁴ x N/m ²)	175	392	192	109	338
Tensile (10 ⁴ x N/m ²)	974	925	1009	933	989
Elong. at Break (%)	623	389	595	794	421
Rebound (%)	43.7	47.4	34.2	55.0	40.6
Compression Set (%) (70 hrs, 150°C)	50	52	65	54	57

25 The results set forth in Table 12 indicate that the carbon
blacks of the present invention can replace the use of blends
of thermal and SRF type carbon blacks to provide a
satisfactory degree of performance characteristics. EPDM
compositions incorporating the carbon black of Example 5 also
30 yield profiles having textured matte finishes when extruded or
shaped.

Table 13 Comparison of EPDM Composition Performance.

Example No.	8	Control A	Control B
Carbon Black Analytical Properties:			
Iodine Number (mg/g)	57.9	35.4	40.0
DBP (cc/100g)	95.8	91.0	111.7
CTAB (m ² /g)	53.1	35.9	44.3
Tint (%)	74.2	57.8	64.6
Mode Stokes Dia. (nm)	140	206	144
Median Stokes Dia. (nm)	180	220	186
M-Ratio (Median/Mode Stokes)	1.29	1.07	1.29
EPDM Composition Performance at 200 phr:			
Viscosity (Pa·S) @ 10 sec ⁻¹	23700	24000	30700
Viscosity (Pa·S) @ 150 sec ⁻¹	3130	2850	3450
Mixing Energy (MJ/m ³)	1581	1252	1561
Extrusion Rate (g/min)	50.6	41.1	44.7
Extrusion Shrinkage (%)	12.5	15.9	18.2
t'90 (min)	9.9	7.41	7.45
ΔL (g·m)	218	299	293
Hardness (Shore A)	79	75	82
E100 (10 ⁴ x N/m ²)	628	493	650
Tensile (10 ⁴ x N/m ²)	1268	1030	1103
Elong. at Break (%)	237	288	184
Rebound (%)	30.5	34.5	31.0
Compression Set (%) (70 hrs, 150°C)	58	50	57

The results set forth in Table 13 indicate that the EPDM composition incorporating the carbon black of the present invention has a higher extrusion rate than the EPDM

compositions incorporating the control carbon black. Thus, EPDM compositions incorporating the carbon blacks of the present invention may be extruded at higher throughputs than EPDM compositions incorporating the control carbon blacks.

The EPDM composition incorporating the carbon black of the present invention also has a lower extrusion shrinkage and therefore will have better dimensional control during th

extrusion operation than the EPDM compositions incorporating the control carbon blacks.

5 These results also indicate that the EPDM composition incorporating the carbon black of the present invention has good overall physical properties. Additionally, the carbon blacks of the present invention have a unique combination of surface area and structure that results in the carbon blacks being useful in preparing EPDM compositions that are to be UHF cured.

10 It should be clearly understood that the forms of the present invention herein described are illustrative only and are not intended to limit the scope of the invention.

CLAIMS

We Claim

1. A carbon black having an I₂No. of 17-23 mg/g and a DBP of 115-150 cc/100g.
- 5 2. The carbon black of claim 1 wherein the I₂No. is 20 mg/g.
3. A carbon black having an I₂No. of 10-19 mg/g, and a DBP of 70-95 cc/100g.
4. The carbon black having an I₂No. of 12-20 mg/g and a
10 DBP of 34-65 cc/100g.
5. The carbon black of claim 4 wherein the I₂No. is 14-18 mg/g and the DBP is 36-55 cc/100g.
6. The carbon black of claim 5 wherein the DBP is 36-42 cc/100g.
- 15 7. The carbon black of claim 5 wherein the DBP is 45-55 cc/100g.
8. A carbon black having an I₂No. of 28-43 mg/g and a DBP of 28-47cc/100g.
9. The carbon black of claim 8 wherein the I₂No. is 30-
20 42 mg/g.
10. A carbon black having an I₂No. of 8-32 mg/g, a DBP of 28-150 cc/100g and an M-Ratio of 1.25-2.00.
11. A carbon black having an I₂No. of 33-70 mg/g, a DBP of 28-60 cc/100g and an M-Ratio of 1.25-2.00.
- 25 12. A carbon black having an I₂No. of 42-50 mg/g, a DBP

of 61-105 cc/100g and an M-Ratio of 1.25-2.00.

13. A carbon black having an I₂No. of 51-62 mg/g, a DBP of 61-125 cc/100g and an M-Ratio of 1.25-2.00.

14. A carbon black having an I₂No. of 63-70 mg/g, a DBP of 61-105 cc/100g and an M-Ratio of 1.25-2.00.

15. A composition of matter comprising a material selected from the group consisting of rubbers and plastics, and a carbon black of claim 1.

16. A composition of matter comprising a material selected from the group consisting of rubbers and plastics, and a carbon black of claim 3.

17. A composition of matter comprising a material selected from the group consisting of rubbers and plastics, and a carbon black of claim 4.

18. A composition of matter comprising a material selected from the group consisting of rubbers and plastics, and a carbon black of claim 8.

19. A composition of matter comprising a material selected from the group consisting of rubbers and plastics, and a carbon black of claim 10.

20. A composition of matter comprising a material selected from the group consisting of rubbers and plastics, and a carbon black of claim 11.

21. A composition of matter comprising a material selected from the group consisting of rubbers and

- plastics, and a carbon black of claim 12.
22. A composition of matter comprising a material selected from the group consisting of rubbers and plastics, and a carbon black of claim 13.
- 5 23. A composition of matter comprising a material selected from the group consisting of rubbers and plastics, and a carbon black of claim 14.

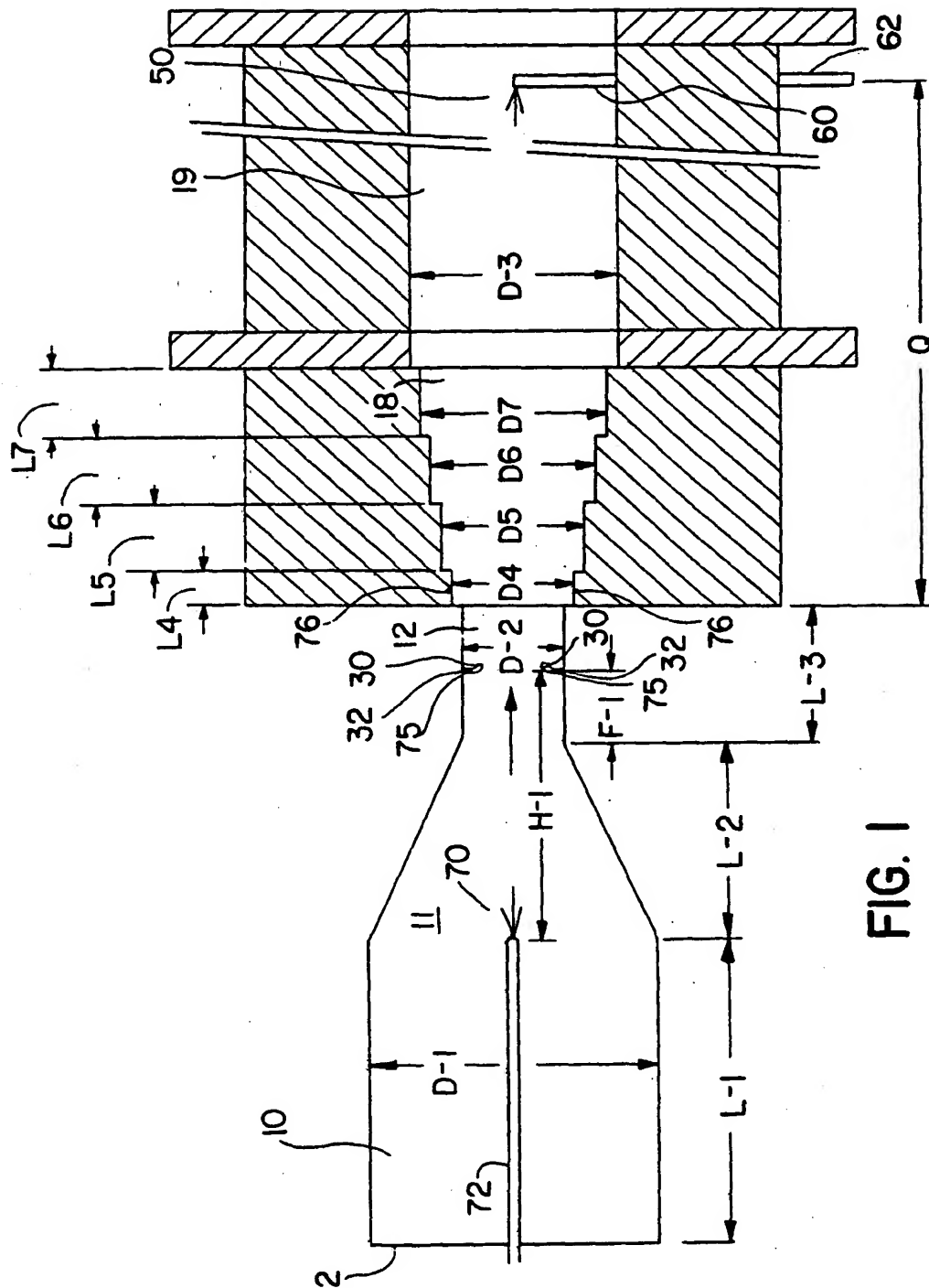
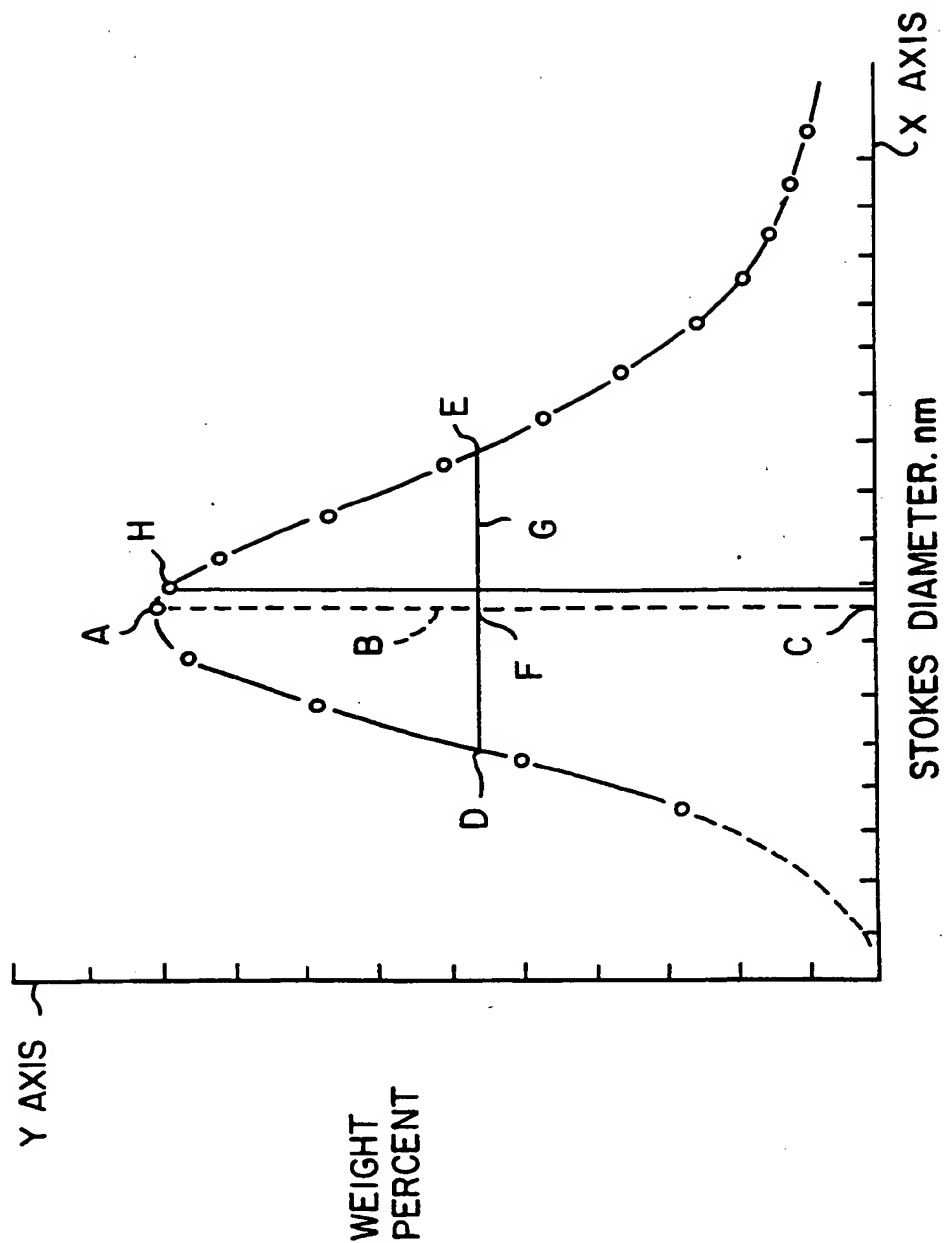


FIG. 1

FIG. 2



SUBSTITUTE SHEET

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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(54) Title: CARBON BLACKS (57) Abstract Novel carbon blacks that impart advantageous properties to rubber and plastic compositions and may be utilized in place of lamp blacks, thermal carbon blacks and blends of carbon blacks. Also disclosed are novel rubber and plastic compositions incorporating the novel carbon blacks which exhibit advantageous combinations of compound processing and physical performance properties.		

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INTERNATIONAL SEARCH REPORT

Inter- national Application No
PCT/US 93/08129

A. CLASSIFICATION OF SUBJECT MATTER
IPC 5 C09C1/50 C08K3/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 C09C C08K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	PATENT ABSTRACTS OF JAPAN vol. 14, no. 137 (C-702)15 March 1990 & JP,A,02 011 664 (ASAHI CARBON) 16 January 1990	1,2,15
A	see abstract ---	3,10,16, 19
X	FR,A,2 064 877 (CONTINENTAL CARBON COMPANY) 23 July 1971	1-3
A	see table II ---	10
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☒ Patent family members are listed in annex.

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- "&" document member of the same patent family

Date of the actual completion of the international search

17 May 1994

Date of mailing of the international search report

0 8. 06. 94

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Van Bellingen, I

INTERNATIONAL SEARCH REPORT

 Inter nal Application No
 PCT/US 93/08129

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DATABASE WPI Week 8942, Derwent Publications Ltd., London, GB; AN 89-306640 & JP,A,1 229 074 (ASAHI CARBON) 12 September 1989	4-6, 17
A	see abstract ---	3, 7, 10, 16, 19
X	DATABASE WPI Week 9106, Derwent Publications Ltd., London, GB; AN 91-41095 & JP,A,2 308 835 (TOYO RUBBER IND.) 21 December 1990	1, 2, 15
A	see abstract ---	3, 10-14, 16, 19-23
P, X	WO,A,93 10194 (CABOT CORPORATION) 27 May 1993	10, 12-14, 19, 21-23
A	see claims 1, 5; table 3 ---	1, 2, 11, 15, 20
A	DATABASE WPI Week 9235, Derwent Publications Ltd., London, GB; AN 92-289009 & JP,A,4 198 273 (FUJIKURA) 17 July 1992 see abstract ---	1-23
A	DATABASE WPI Week 8516, Derwent Publications Ltd., London, GB; AN 85-095390 & JP,A,60 042 439 (TOKAI CARBON) 6 March 1985 see abstract ---	1, 2, 10-15, 19-23
A	DATABASE WPI Week 9139, Derwent Publications Ltd., London, GB; AN 91-286149 & JP,A,3 190 945 (YOKOHAMA RUBBER) 20 August 1991 see abstract ---	11, 14, 20, 23
3 A	EP,A,0 020 889 (DEGUSSA) 7 January 1981 see claim 1 --- -/--	11

INTERNATIONAL SEARCH REPORT

Inter nal Application No
PCT/US 93/08129

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US,A,3 961 902 (J. W. VANDERVEEN) 8 June 1976 see column 3, line 55 - line 62 ---	11
A	US,A,4 601 892 (C. J. HART ET AL.) 22 July 1986 see column 8, line 50 - line 53 -----	10

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 93/08129

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

- | | |
|-------------------------------------|---------------------|
| 1. Claims 1,2,15 and 10 (partially) | 6. Claims 11 and 20 |
| 2. Claims 3,16 and 10 (partially) | 7. Claims 12 and 21 |
| 3. Claims 4-7,17 and 10 (partially) | 8. Claims 13 and 22 |
| 4. Claims 8,9 and 18 | 9. Claims 14 and 23 |
| 5. Claims 10 and 19 | |

1. ☒ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☒ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 93/08129

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